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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/086,025	02/28/2002	Marc R. Anderson	286697-00005	7853

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EXAMINER

SODERQUIST, ARLEN

ART UNIT	PAPER NUMBER
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1743

DATE MAILED: 10/24/2005

Please find below and/or attached an Office communication concerning this application or proceeding.

## Office Action Summary

**Application No.**

10/086,025

**Applicant(s)**

ANDERSON ET AL.

**Examiner**

Arlen Soderquist

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-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

**Period for Reply**

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

**Status**

- 1) ☒ Responsive to communication(s) filed on 25 July 2005.
- 2a) ☐ This action is **FINAL**. 2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

**Disposition of Claims**

- 4) ☒ Claim(s) 109-113, 118-120, 122 and 123 is/are pending in the application.
- 4a) Of the above claim(s) \_\_\_\_\_ is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 109-112, 118-120 and 122 is/are rejected.
- 7) ☒ Claim(s) 113 and 123 is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_\_ are subject to restriction and/or election requirement.

**Application Papers**

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on \_\_\_\_\_ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.  
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).  
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

**Priority under 35 U.S.C. § 119**

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All b) ☐ Some \* c) ☐ None of:
- ☐ Certified copies of the priority documents have been received.
  - ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.
  - ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

**Attachment(s)**

- ☒ Notice of References Cited (PTO-892)
- ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
- ☐ Information Disclosure Statement(s) (PTO-1449 or PTO/SB/08)  
Paper No(s)/Mail Date \_\_\_\_\_.
- ☐ Interview Summary (PTO-413)  
Paper No(s)/Mail Date. \_\_\_\_\_.
- ☐ Notice of Informal Patent Application (PTO-152)
- ☐ Other: \_\_\_\_\_.

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1. A request for continued examination under 37 CFR 1.114, including the fee set forth in 37 CFR 1.17(e), was filed in this application after final rejection. Since this application is eligible for continued examination under 37 CFR 1.114, and the fee set forth in 37 CFR 1.17(e) has been timely paid, the finality of the previous Office action has been withdrawn pursuant to 37 CFR 1.114. Applicant's submission filed on July 25, 2005 has been entered.
2. Claim 119 is objected to because of the following informalities: in claim 109, lines 12-13 "such that the sample" was apparently intended; in claim 119, line 7 "the equilibrated mixture" was apparently intended. Applicant is encouraged to review the claims for similar informalities. Appropriate correction is required.
3. Claims 109-124 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. In claim 118 it is not clear if the plurality of spike sources are in addition to the spike reservoir of claim 109 or if they are a further definition of the spike reservoir. In claim 110, it appears that the sample extraction apparatus should be required to deliver or transfer the extracted sample to the mixer of claim 109. In other words the limitation of claim 110 is lacking a structural connection to the other elements of the device. Claim 112 also lacks a structural connection with the other elements of the system.
4. Claims 119-124 are rejected under 35 U.S.C. 112, second paragraph, as being incomplete for omitting essential steps, such omission amounting to a gap between the steps. See MPEP § 2172.01. The omitted steps are: that the spike concentration is about the same concentration as the analyte in the sample. In the discussion of the basis for claim 119 in the paper filed April 20, 2004, applicant points to the paragraph starting on page 30, line 19. Two sentences of that paragraph from page 30, lines 22-27 are reproduced below.

"It is necessary in embodiments of the invention that the relative concentration of the spike for any specie for which monitoring is accomplished be in the same general range as the expected concentration in the sample. If the spike concentration is either much too high or much too low, then the mass spectrometer will not be able to provide a ratio of isotopes with adequate resolution to determine the sample concentration."

It is clear from this section that a specific relationship is required between the spike concentration and the concentration of the monitored specie in the sample for the method to

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provide a ratio with adequate resolution as in claim 121. Thus this condition is required or necessary for the method to perform its intended purpose.

5. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.
  2. Ascertaining the differences between the prior art and the claims at issue.
  3. Resolving the level of ordinary skill in the pertinent art.
  4. Considering objective evidence present in the application indicating obviousness or nonobviousness.
1. Claims 109-112, 118-120 and 122 are rejected under 35 U.S.C. 103(a) as being unpatentable over Marchante-Gayon, Rottmann or Viczian in view of Waygood and Godec or May and Stewart or Schramel (last five newly cited and applied).

In the paper Marchante-Gayon presents a study of random and systematic errors for the determination of molybdenum by inductively coupled plasma mass spectrometry using online isotope dilution analysis. Reverse isotope dilution analysis was applied for the determination of the concentration of a  $^{95}\text{Mo}$  enriched spike and the procedure was automated using an auto sampler where natural Mo standards and samples were alternatively mixed online with the spike solution. A new equation is proposed for the online mixing of samples and spike using the autosampler. The measurement of a natural Mo standard between the samples makes possible to perform isotope dilution analysis referring the results to the natural Mo standard regardless of the concentration of the spike. The effects of both systematic and random errors were examined and the error theory was applied for the accurate determination of Mo in biological materials by ID-ICP-MS. On page 86, the second and third paragraphs teach, two solutions having molybdenum at concentrations of about 1  $\mu\text{g/g}$  produced by diluting a first solution to the desired concentration. The second solution is the enriched isotopic spike solution. The last paragraph of

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page 86 teaches this spike added online with the help of an autosampler, a two channel pump and a T piece placed before the nebulizer. Marchante-Gayon does not teach all of the system components.

In the paper Rottmann presents the development of an online isotope dilution technique with HPLC/ICP-MS for the accurate determination of elemental species. An online isotope dilution technique was developed for use with a HPLC system (HPLC) coupled to an inductively coupled plasma mass spectrometer (ICP-MS). With this method it is possible to characterize elemental species at low concentration levels and to quantify them accurately. The possibilities of this method are shown using the samples of the determination of the interactions of different molecular weight fractions of dissolved organic matter (DOM) with copper and molybdenum in a natural water sample. Page 3711 teaches that the spike solution is prepared by diluting a commercially available standard solution or stock solutions prepared from isotopically enriched solids. The last full paragraph of the page teaches mixing the spike with the sample online through a Y-junction. Rottmann does not teach all of the system components.

In the paper Viczian teaches on-line isotope dilution and sample dilution by flow injection and inductively coupled plasma mass spectrometry. A systematic investigation was made to demonstrate the applicability of a flow injection system for online isotope dilution and online sample dilution, as obtained by merging the sample solution with the spike solution or with the diluent, respectively. The effect of the sample to spike ratio on the precision and accuracy was examined, and the advantages and limitations of the proposed technique are demonstrated. A plurality of enriched spike concentrations is taught on page 127 in the second paragraph of the "On-line Isotope Dilution and Spike to Sample Ratio" heading. Viczian does not teach all of the system components.

In the paper Waygood teaches online, real time, continuous analytical and closed loop control of process solutions for electroplating using ICP chemistry. The process of continuously electroplating steel coil with either tin or chromium, mainly for the canmaking industry, has gradually increased in speed. The strip is now processed through the various baths at speeds up to 600 m/min. Producing material at these speeds demands extremely close tolerances upon the various process solutions. The authors have developed an automated online, real time analytical system for these process solutions. The system consists of: a large bore sampling pipeline

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network continuously delivering process solutions to the laboratory, an industrial robot for sampling and manipulating the samples and standard solutions, an autosampler/wash station for presenting samples to the spectrometer, a fully computerized ICP spectrometer for elemental analysis of the various process solutions. Software, specially developed is used to: interface the robot to the sampling hardware and ICP, interface the ICP analysis results to the data management system and, where appropriate, achieve closed loop control of process solution concentrations, and introduce manual samples while carrying out routine automated analysis. A full analysis of 12/13 process lines is achieved every 14 minutes, and restandardization is normally carried out automatically once every 8 hours. Current routine maintenance of the system requires ~30 min per 24 hours.

In the paper Godec discusses the verification of analytical ultrapure water instrumentation performance using an automated standard dilution apparatus. In the past 15 years, improvements in industrial water purification and measurement technologies have been driven by increasing high purity requirements for Si wafer rinse water. The typical levels of impurities of this water are often pushing or exceeding the limits of detection for both online analyzers and off-line laboratory analyzers. Analysis must often be done using sample pre-concentration methods. Water collection methods for off site analysis require great efforts to prevent sample contamination. Trace level analysis can also be altered by sample matrix problems, instrument recovery problems, or unstable instrument baselines and backgrounds. Online instrumentation usually lacks built in quality control methods. For these and other reasons it is often difficult for the water plant operator to assess the accuracy of either online or off-line analytical measurements. They built a device that can be placed before an analyzer to accurately add known trace levels of compounds to the instrument sample stream. The method overcomes the limitations and difficulties of preparing low level standards. Standard solutions in the part per million ranges of contaminants are prepared in vials. The vial solutions are dynamically diluted into the sample stream using a syringe pump. Dilution ratios 1:10,000-1:5,000,000 are easily achieved. This paper addresses the principles of standard addition, methods to evaluate analyzer suitability, and provides an example of the use of this device to resolve a detector problem at a semiconductor site.

In the paper May discusses the CETAC ADX-500 autodiluter system and its dilution

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performance with the ELAN 6000 ICP-MS and ELAN software. The CETAC ADX-500 autodiluter system was tested with ELAN v. 2.1 software and the ELAN 6000 ICP-MS instrument to determine online automated dilution performance during analysis of standard solutions containing nine analytes representative of the mass spectral range (mass 9 to mass 238). Two or more dilution schemes were tested for each of 5 test tube designs. Dilution performance was determined by comparison of analyte concentration means of diluted and nondiluted standards. Accurate dilutions resulted with one syringe pump addition of diluent in small diameter round-bottomed (13 mm OD) or conical-tipped (18 mm OD) tubes and one or more syringe pump additions in large diameter (28 mm OD) conical-tipped tubes. Inadequate dilution mixing which produced high analyte concentration means was observed for all dilutions conducted in flat-bottomed tubes, and for dilutions requiring multiple syringe additions of diluent in small diameter round-bottomed and conical tipped tubes. Effective mixing of diluted solutions depends largely upon tube diameter and liquid depth: smaller tube diameters and greater liquid depth resulted in ineffective mixing, whereas greater tube diameter and shallower liquid depth facilitated effective mixing. Two design changes for the autodiluter were suggested that would allow effective mixing to occur using any dilution scheme and tube design.

In the paper Stewart presents investigations into chromium speciation by electrospray mass spectrometry. Chromium is an element whose toxicity is oxidation state dependent. Chromium(VI) is considered toxic to humans whereas Cr(III) is considered to be an essential nutrient. It therefore becomes important to be able to distinguish between the two oxidation states in solution. Electrospray mass spectrometry (ESMS) has the potential to provide useful information on chromium species in solution samples. The various solution forms of both oxidation states may be observed directly and are presented. Other more uncommon species of both oxidation states such as polymeric Cr(III) and the polyanion trichromate are discussed. Finally, the ability of ESMS to monitor other aspects of the solution chemistry of both chromium oxidation states is explored.

In the paper Schramel presents analysis of metal species by using electrospray ionization mass spectrometry and capillary electrophoresis-electrospray ionization mass spectrometry. Metal speciation was carried out by online hyphenation of capillary electrophoresis (CE) with mass spectrometry (MS) via an electrospray ionization (ESI) interface. The commercially

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available interface was hardly able to produce stable electrospray conditions over an extended period of time, mainly caused by an insufficient positioning of the CE capillary inside the ESI stainless steel tip. A device was developed, which allowed an infinitely variable adjustment of the capillary. The optimum position for stable electrospray conditions was set to 0.4-0.7 mm outside the ESI tip. Off-line ESI-MS studies of free metal ions [Cu(II)], metal ion-containing complexes [CuEDTA, Me<sub>3</sub>SbCl<sub>2</sub>] and covalent organometallic compounds (selenocystamine, selenomethionine) were carried out to assess the suitability of the technique for metal speciation. The usefulness of ESI-MS as a detection method largely depends on the stability of the analyzed species. Inorganic species (i.e. metal ions) alter their composition when being electrosprayed. Parts of the weakly complexing ligands will be exchanged by solvent molecules, mainly originating from the sheath liquid. The destruction of ion-solvent clusters by heating, collision-induced decomposition or use of a sheath gas may lead to charge reduction. of transition metal ions. Organometallic complexes with strongly complexing ligands remain intact, while those with weakly complexing ligands suffer from the same disadvantages as inorganic species. ESI-MS is best suited for the speciation of covalent organometallic compounds. The ionization process does not alter their structure and they will mostly be detected as singly charged molecular ions.

It would have been obvious to one of ordinary skill in the art at the time the invention was made to incorporate total automation as taught by Waygood and the dilution means as taught by Godec or May to automatically prepare the spike as taught by Marchante-Gayson, Rottmann or Viczian because of the advantages for automation taught by Waygood and Godec or May or generally known in the field. It should be noted that claims 109 and 119 do not require the connection of the analyzer to the process solution. It would have been obvious to one of ordinary skill in the art at the time the invention was made to use an atmospheric pressure ionization technique as taught by Stewart or Schramel for the mass spectrometric detection in the device and methods taught by Marchante-Gayson, Rottmann or Viczian because of the automation advantages taught for speciation as taught by Stewart and Schramel.

6. Claims 113 and 123 are objected to as being dependent upon a rejected base claim, but would be allowable if rewritten in independent form including all of the limitations of the base



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claim and any intervening claims. The art of record fails to teach the combination of structure found in claim 113 or the multiple dilutions of the spike as found in claim 123.

7. Applicant's arguments with respect to the claims have been considered but are moot in view of the new ground(s) of rejection.

8. The prior art made of record and not relied upon is considered pertinent to applicant's disclosure. The additionally cited art relates to dilution apparatus and atmospheric pressure ionization.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Arlen Soderquist whose telephone number is (571) 272-1265. The examiner's schedule is variable between the hours of about 6:30 AM to about 5:00 PM on Monday through Thursday and alternate Fridays.

A general phone number for the organization to which this application is assigned is (571) 272-1700. The fax phone number to file official papers for this application or proceeding is (571) 273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).



Arlen Soderquist  
Primary Examiner  
October 17, 2005